Name (Section 102)

September 17, 2002

Lab #1

Recrystallization and Melting Points

Abstract:

Benzoic Acid is recrystallized with a 41% recovery using 95% ethanol and water as the mixed-solvent. Benzoic acid is also recrystallized with a 79% recovery using water as the solvent. The product is a white crystalline solid (MP 114-122 °C and 121-127 °C, respectively) after recrystallization.

Introduction:

This experiment was conducted in order to explore the methods of recrystallization and in order to determine the melting points of various solids. Recrystallization involves dissolution of a solid in a solvent at elevated temperatures and the reformation of the crystals as the solution cools, allowing for impurities to remain in the solution (Gilbert and Martin 89). This technique is important to chemistry because it is one of the most often used methods for purification of solids, allowing one to achieve one of the highest states of purity. Once a solid has been recrystallized, it is important to determine the purity of the recrystallized solid. An easy way to do this is by taking the melting point of the solid.

Materials and Methods:

Recrystallization of Benzoic Acid was performed according to the method of Gilbert and Morgan (2001, section 3.2). Instead of using water to recrystallize benzoic acid, ethanol was used in 3.2B part 1. The solution was also decolorized twice instead of

once. Once the solution had cooled and no crystals appeared to form, 20mL of water was added and allowed to boil, creating a mixed-solvent. The solution was then allowed to cool and recrystallized. The procedure then continued as described in Gilbert and Martin. Acetanilide, Naphthalene, and an unknown compound were not recrystallized like the procedure in the book described. Because the recrystallization of benzoic acid in part 1 turned out to be a mixed solvent crystallization, section 5 was performed only using water as the solvent, instead of a mixed solvent as suggested. It was observed that the benzoic acid began to recrystallize as soon as it was taken off the heat after it was initially dissolved. Because of this, 25mL of water was added to the solution and the benzoic acid was allowed to dissolve again before it was recrystallized. Determination of melting points was performed according to the method of Gilbert and Martin as well (2001, section 3.3). The thermometers were calibrated before the lab was started so that portion of the procedure was not performed. Rather the values from the calibration were obtained by the TA and distributed.

Results:

The recrystallized benzoic acid appeared as a white (with a slight purple tint), powdery solid weighing .41g when it was recrystallized using 95% ethanol and water. The percent recovery was 41%. The melting point of the crude benzoic acid was 118-122 C, while the melting point of the recrystallized benzoic acid was 114-122 C. When recrystallized using just water as the solvent the benzoic acid appeared as a white, powdery solid weighing .87g. The percent recovery was 79%. The melting point of the crude benzoic acid was 118-122 C, while the melting point of the recrystallized benzoic acid was 121-127 C.

Discussion:

95% ethanol was used in the first part of this lab because when the solvent selection was performed, the results indicated that benzoic acid was insoluble in water at both room temperature and when it was hot. These results could have been an error caused maybe by not allowing the benzoic acid to sit in the hot solvent long enough for it to dissolve. Because of this fact, ethanol was used as the solvent in the first part of the experiment. This caused a few problems, most importantly was the fact that the solution was not recrystallizing once it began to cool after decolorization and filtration. This is the reason that water was added to the solution. By adding water to the solution and allowing it to boil more (to boil off some of the ethanol) a mixed-solvent was formed.

Additionally, decolorization of the solution proved to be a problem. A large quantity of carbon had to be used in order to decolorize the benzoic acid/ethanol solution. Once decolorization failed the first time around, the solution had to be decolorized again using a much larger quantity of carbon. This proved to be messy and made hot filtration more problematic because there was so much carbon to filter out, the filtering went slow and it was hard to keep the solution hot. The percent recovery for this part of the lab could have been so low (41%) because of this fact. It may have been the case that some of the solution could have cooled enough during the hot filtration to allow for some of the benzoic acid to recrystallize and get filtered out from the solution at this point. This would explain why there was not a high percent recovery at the end of the experiment when the recrystallized benzoic acid was massed.

The melting points of both sets of recrystallized benzoic acids were similar to the expected value. The melting point of benzoic acid is approximately 122 C (2002, Gilbert and Martin CD-Rom, MSDS Data). The melting points of the recrystallized benzoic acid were 114- 122 C and 121-127 C, both encompassing the expected value and allowing one to believe that the sample was close to pure, which was the goal of recrystallization.

References/Literature Cited:

Gilbert, J. C.; Martin, S. F. *Experimental Organic Chemistry, 3 rd Ed.*; Brooks/Cole-Thompson Learning: Pacific Grove, 2002, Chapter 3, 88-113.

http://www.emory.edu/CHEMISTRY/mccormick/powerpoint/sld003.htm

Figures/Tables/Data:

Table 1: MSDS Data on Various Solvents

Solvent	Physical State	Color	Melting Point (C)	Boiling Point (C)
Benzoic Acid	Solid	White	122	249
Acetanilide	Solid	White	114	304
Naphthalene	N/A	N/A	80.5	218
Resorcinol	Solid	White	110-113	280

Table 2: Solubility of Various Solutes at Room Temperature and Hot Temperatures

Solute	Unknown A	Benzoic Acid	Acetanilide	Naphthalene	Resorcinol
Water Room	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
Water Hot	Soluble	Insoluble	Soluble	Soluble	Soluble
Ethanol Room	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
Ethanol Hot	Soluble	Soluble	Soluble	Soluble	Soluble
Petroleum Ether Room	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
Petroleum Ether Hot	Soluble	Insoluble	Insoluble	Soluble	Soluble

Table 3: Benzoic Acid Recrystallization using Ethanol and Water as the Solvent: Melting Points, Percent Recovery, and Weights

Weight Of Benzoic Acid prior to recrystallization:	1.01 g
Weight of Benzoic Acid after recrystallization:	.41 g
Weight Watch glass:	44.24 g
Weight of Watch glass and Recrystallized Benzoic Acid:	44.65 g
Volume Ethanol Added: Volume of Water Added:	12.0 mL 20.0 mL
Melting Point of crude Benzoic Acid:	118-122 C
Melting Point of Recrystallized Benzoic Acid:	114-122 C
Percent Recovery:	40.6%

Table 4: Benzoic Acid Recrystallization using Water as the Solvent: Melting Points, Percent Recovery, and Weights

Weight of Benzoic Acid prior to recrystallization:	1.10 g
Weight of Benzoic Acid after recrystallization:	.87 g

Weight of watch glass:	44.24 g
Weight of watch glass and recrystallized Benzoic Acid:	45.11 g
Volume of Water added:	35.0 mL
Melting Point of Crude Benzoic Acid:	118-122 C
Melting Point of Recrystallized Benzoic Acid:	121-127 C
Percent Recovery:	79.1%

Table 5: Melting Point deviations from expected value. Calibration

Compound	Literature Melting Point (C)	Actual Melting Point (C)
3 Phenylpropionic Acid	48.6	43-45
Acetamide	82.3	69-71
Benzamide	133	103-117
Ice	0	3

Table 6: Melting points of various compounds determined experimentally.

Compound	Melting Point (C)
Acetanilide	101-106.5
Unknown A	100-107
Acetanilide + Unknown A	100-104
Crude Benzoic Acid	118-122

Questions: